


Electrical-Field Activated Sintering and Forming of Micro-Components

Muhammad Bin Zulkipli



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Dedication

The author wished to dedicate the thesis to his wife Ilyana Janis and families.



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Abstract

As the demand for miniature products has increased significantly, so also has the need for these products to be produced in a rapid, flexible and cost efficient manner. The application of electroplasticity shows significant potential to produce the components by using powder materials. Nevertheless, previous research has shown that there are still significant challenges to be met in order to achieve increased relative densification of product samples and simplification of the processes. The process concept in this study comprises the combination of electrical-field activated sintering and forming processes. Therefore, the aims of the research were to develop the process concept for the manufacture of micro-components and to design the die sets along with other tooling for machine setup to enable the forming of micro-components from powder materials. A comprehensive literature review on micro-manufacturing, size effects, powder metallurgy and the electroplasticity process has been conducted. The development of the die sets for the process has been described, followed by a series of experiments. The FE thermal-electrical analysis was also carried out to study the heating flows of the die sets development during the process. In this research, titanium (Ti) and titanium tin alloy (90Ti10Sn) have been selected for the main powder materials tested for both vacuum and open-air process environment by using a Gleeble® 3800 testing system and Projection Welding machine respectively. Meanwhile, for the additional experiment, copper (Cu) has been selected to be tested in the open-air process environment by using a Projection Welding machine with die sets prepared by the Micro-FAST project. Based on the data collected, this efficient process has the potential to produce components with a high relative density of around 98%. Changes of the particles concerning deformation and breaking are crucial in the course of achieving the densification which differs from a conventional sintering process.

Published Research Papers and Posters

a) Research Papers:

1. **Muhammad Bin Zulkipli**, Yi Qin, Kunlan Huang, Hasan Hazma Hijji, Yihui Zhao and Jie Zhao (6th to 9th August 2015), "*Forming of titanium and titanium alloy miniature-cylinder by electrical- field activated powder sintering and forming*". 4th International Conference on New Forming Technology (ICNFT2015) – Glasgow, Scotland, United Kingdom. (<http://dx.doi.org/10.1051/mateconf/20152110006>).
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4. Hasan Hijji, Yi Qin, Kunlan Huang, **Muhammad Bin Zulkipli**, Song Yang and Jie Zhao (6th to 8th September 2016), "*Forming Alumina (Al₂O₃) by Micro-FAST*". 14th International Conference on Manufacturing Research (ICMR2016) – Loughborough, England, United Kingdom. (<http://ebooks.iospress.nl/volumearticle/44320>).
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Chapter 1: Introduction



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

1.1. Research Background

In general demands for micro-electromechanical systems (MEMS) and micro-products have significantly increased due to the fast growth that can be seen in the field of biomedicine, telecommunications and automotive engineering. For example, the global industry association Semiconductor Equipment and Materials International (SEMI) has released the sales forecast for semiconductor equipment which had a market value of \$37 billion in 2015 to approximately \$38 billion in 2016 and enjoys an active growth in the global market of 1.4% [1]. Besides typical MEMS manufacturing methods, significant efforts have been made recently either to scale down traditional processes or to develop the new ones for the manufacture of micro- and nano-products [2]–[5]. For instance, the production of micro-gears which are significant actuating and transmission components used widely in MEMS and micro-mechanical-systems (MMS), is often enabled through ultra-precision machining or lithographic techniques especially LIGA processes [6], [7]. Micro-forming also plays important roles for manufacturers of millions of micro screws through upsetting and rolling every year [8]–[10]. In addition to that, micro-extrusion has been well investigated where it is capable of extruding copper pins in the laboratory with a shaft diameter of 0.5 to 0.8 mm and a wall thickness of 50 to 125 μm [11], [12]. Therefore, based on the previous research and experiment results there is no doubt that micro-products with high precision and accuracy can be produced, nevertheless concerning cycle time and cost for high production, there are still improvements to be made.

In micro-forming, the limits of the process are influenced largely by the workpiece's dimension. This phenomenon is being commonly referred as the size effects. The size effects that exist in metallic materials can be described as grain and feature/specimen size effects. According to Armstrong [13], grain size effect can be represented by the Hall-Petch law which states that the material strengthens as the grain size decreases. Meanwhile for feature/specimen size effects observed when the miniaturisation of the part occurs resulting in the decline of the flow stress. In this feature/specimen size effect, the workpiece cannot be considered as an isotropic continuum due to the huge share of the volume occupied by individual grains with different orientations if the microstructure, surface topology and tools of the workpiece used in the process remain unchanged [12], [14]. It was found that by

References

- [1] "Semiconductor Equipment Sales Forecast: \$37 Billion in 2015 and \$38 Billion in 2016," *SEMI Organization*, 2015. [Online]. Available: <http://www.semi.org/en/node/60181>. [Accessed: 29-Sep-2016].
- [2] C. K. Malek and V. Saile, "Applications of LIGA technology to precision manufacturing of high-aspect-ratio micro-components and -systems: a review," *Microelectronics J.*, vol. 35, no. 2, pp. 131–143, Feb. 2004.
- [3] S. Y. Liang, "Mechanical machining and metrology at micro/nano scale," in *Proc. of SPIE - The International Society for Optical Engineering, Third International Symposium on Precision Mechanical Measurements*, 2006, vol. 6280, pp. 628002–628008.
- [4] Y. Qin, "Overview of Micro-Manufacturing," in *Micro-Manufacturing Engineering and Technology*, Elsevier Inc., 2010, pp. 1–23.
- [5] Y. Qin, A. Brockett, Y. Ma, A. Razali, J. Zhao, C. Harrison, W. Pan, X. Dai, and D. Loziak, "Micro-manufacturing: research, technology outcomes and development issues," *Int. J. Adv. Manuf. Technol.*, vol. 47, no. 9–12, pp. 821–837, 2010.
- [6] R. Ruprecht, T. Benzler, T. Hanemann, K. Müller, J. Konys, V. Piotter, G. Schanz, L. Schmidt, A. Thies, H. Wöllmer, and J. Haußelt, "Various replication techniques for manufacturing three-dimensional metal microstructures," *Microsyst. Technol.*, vol. 4, no. 1, pp. 28–31, 1997.
- [7] S. C. H. Thian, Y. Tang, J. Y. H. Fuh, Y. S. Wong, L. Lu, and H. T. Loh, "Micro-rapid-prototyping via multi-layered photo-lithography," *Int. J. Adv. Manuf. Technol.*, vol. 29, no. 9–10, pp. 1026–1032, 2006.
- [8] Y. Qin, "Micro-forming and miniature manufacturing systems — development needs and perspectives," *J. Mater. Process. Technol.*, vol. 177, no. 1–3, pp. 8–18, Jul. 2006.
- [9] A. Hannewald, "Micro-screws from the rotary press," *Umformtechnik*, vol. 2, pp. 28–29, 2002.
- [10] F. Vollertsen, Z. Hu, H. S. Niehoff, and C. Theiler, "State of the art in micro forming and

- investigations into micro deep drawing," *J. Mater. Process. Technol.*, vol. 151, no. 1–3, pp. 70–79, Sep. 2004.
- [11] U. Engel and E. Egerer, "Basic Research on Cold and Warm Forging of Microparts," *Key Eng. Mater.*, vol. 233–236, pp. 449–456, 2003.
- [12] M. Geiger, M. Kleiner, R. Eckstein, N. Tiesler, and U. Engel, "Microforming," *CIRP Ann. - Manuf. Technol.*, vol. 50, no. 2, pp. 445–462, Jan. 2001.
- [13] R. W. Armstrong, "On size effects in polycrystal plasticity," *J. Mech. Phys. Solids*, vol. 9, no. 3, pp. 196–199, Jul. 1961.
- [14] N. Tiesler and U. Engel, "Microforming - effects of miniaturisation," in *Proceedings of 8th International Conference on Metal Forming*, 2000, pp. 355–360.
- [15] E. Uhlmann, G. Spur, N. A. Daus, and U. Doll, "Application of μ -EDM in the machining of micro structured forming tools," *Tech. Pap. - Soc. Manuf. Eng. AD*, no. 285, pp. 1–11, 1999.
- [16] E. Uhlmann, S. Piltz, and U. Doll, "Machining of micro/miniature dies and moulds by electrical discharge machining—Recent development," *J. Mater. Process. Technol.*, vol. 167, no. 2–3, pp. 488–493, Aug. 2005.
- [17] R. Orrù, R. Licheri, A. M. Locci, A. Cincotti, and G. Cao, "Consolidation/synthesis of materials by electric current activated/assisted sintering," *Mater. Sci. Eng. R Reports*, vol. 63, no. 4–6, pp. 127–287, 2009.
- [18] F. F. Lange, "Densification of powder compacts: An unfinished story," *J. Eur. Ceram. Soc.*, vol. 28, no. 7, pp. 1509–1516, Jan. 2008.
- [19] M. Zadra, F. Casari, L. Girardini, and A. Molinari, "Spark plasma sintering of pure aluminium powder: mechanical properties and fracture analysis," *Powder Metall.*, vol. 50, no. 1, pp. 40–45, 2007.
- [20] N. P. Mahalik, "Introduction," in *Micromanufacturing and Nanotechnology*, Berlin/Heidelberg: Springer-Verlag, 2006, pp. 1–468.
- [21] J. Jeswiet, M. Geiger, U. Engel, M. Kleiner, M. Schikorra, J. Duflou, R. Neugebauer, P. Bariani, and S. Bruschi, "Metal forming progress since 2000," *CIRP J. Manuf. Sci. Technol.*, vol. 1, no. 1, pp. 2–17, 2008.

- [22] J. Jeswiet and M. Hauschild, "EcoDesign and future environmental impacts," *Mater. Des.*, vol. 26, no. 7, pp. 629–634, Jan. 2005.
- [23] S. S. Dimov, E. B. J. P. Brousseau, R. Minev, and S. Bigot, "Micro- and nano-manufacturing: Challenges and opportunities," *J. Mech. Eng. Sci.*, vol. 226, pp. 3–15, 2011.
- [24] U. Engel and R. Eckstein, "Microforming—from basic research to its realization," *J. Mater. Process. Technol.*, vol. 125–126, pp. 35–44, Sep. 2002.
- [25] M. Koç and T. Özel, "Fundamentals of Micro-Manufacturing," in *Micro-Manufacturing: Design and Manufacturing of Micro-Products*, John Wiley & Sons, Inc., 2011, pp. 1–23.
- [26] H. Ike and M. Plancak, "Coining process as a means of controlling surface microgeometry," *J. Mater. Process. Technol.*, vol. 80–81, pp. 101–107, Aug. 1998.
- [27] Y. Saotome and H. Iwazaki, "Superplastic backward microextrusion of microparts for micro-electro-mechanical systems," *J. Mater. Process. Technol.*, vol. 119, no. 1–3, pp. 307–311, Dec. 2001.
- [28] A. R. Razali, "High-precision, high speed strip feeding in micro-forming," University of Strathclyde, 2010.
- [29] A. R. Razali and Y. Qin, "A review on micro-manufacturing, micro-forming and their key issues," *Procedia Eng.*, vol. 53, pp. 665–672, 2013.
- [30] P. Rougeot, S. Regnier, and N. Chaillet, "Forces analysis for micro-manipulation," *2005 Int. Symp. Comput. Intell. Robot. Autom.*, pp. 105–110, 2005.
- [31] F. Arai, D. Ando, T. Fukuda, Y. Nonoda, and T. Oota, "Micro manipulation based on micro physics-strategy based on attractive force reduction and stress measurement," *Proc. 1995 IEEE/RSJ Int. Conf. Intell. Robot. Syst. Hum. Robot Interact. Coop. Robot.*, vol. 2, pp. 236–241, 1995.
- [32] F. Arai and T. Fukuda, "A new pick up and release method by heating for micromanipulation," *Proc. IEEE Tenth Annu. Int. Work. Micro Electro Mech. Syst. An Investig. Micro Struct. Sensors, Actuators, Mach. Robot.*, no. i, pp. 1–6, 1997.
- [33] R. S. Fearing, "Survey of sticking effects for micro parts handling," *Proc. 1995 IEEE/RSJ Int. Conf. Intell. Robot. Syst. Hum. Robot Interact. Coop. Robot.*, vol. 2,

1995.

- [34] J. T. Feddema, P. Xavier, and R. Brown, "Micro-assembly planning with van der Waals force," *Proc. 1999 IEEE Int. Symp. Assem. Task Plan. (Cat. No.99TH8470)*, no. July, pp. 32–38, 1999.
- [35] J. Tomas, "Adhesion of ultrafine particles—A micromechanical approach," *Chem. Eng. Sci.*, vol. 62, no. 7, pp. 1997–2010, Apr. 2007.
- [36] R. B. Aronson, "The New World of Micromanufacturing," *Manuf. Eng.*, vol. 140, p. 4, 2003.
- [37] R. B. Aronson, "Micromanufacturing is growing," *Manuf. Eng.*, vol. 132, p. 4, 2004.
- [38] Y. Kibe, Y. Okada, and K. Mitsui, "Machining accuracy for shearing process of thin-sheet metals—Development of initial tool position adjustment system," *Int. J. Mach. Tools Manuf.*, vol. 47, no. 11, pp. 1728–1737, 2007.
- [39] B.-Y. Joo, S.-H. Rhim, and S.-I. Oh, "Micro-hole fabrication by mechanical punching process," *J. Mater. Process. Technol.*, vol. 170, no. 3, pp. 593–601, Dec. 2005.
- [40] G.-L. Chern and Y. Chuang, "Study on vibration-EDM and mass punching of micro-holes," *J. Mater. Process. Technol.*, vol. 180, no. 1–3, pp. 151–160, Dec. 2006.
- [41] Y. Okazaki, N. Mishima, and K. Ashida, "Microfactory—Concept, History, and Developments," *J. Manuf. Sci. Eng.*, vol. 126, no. 4, p. 837, 2004.
- [42] L. Alting, F. Kimura, H. N. Hansen, and G. Bissacco, "Micro engineering," *CIRP Ann. - Manuf. Technol.*, vol. 52, no. 2, 2003.
- [43] B. Nau, A. Roderburg, and F. Klocke, "Ramp-up of hybrid manufacturing technologies," *CIRP J. Manuf. Sci. Technol.*, vol. 4, no. 3, pp. 313–316, Jan. 2011.
- [44] D. F. Heaney, "Mass production of micro-components utilising lithographic tooling and injection molding technologies," in *Proceedings of 1st International Conference on Micro-Manufacturing*, 2006, pp. 280–283.
- [45] J. A. Palmer, J. D. Williams, T. Lemp, T. M. Lehecka, F. Medina, and R. B. Wicker, "Advancing three-dimensional MEMS by complimentary laser micro manufacturing," in *Proceedings SPIE 6109, Micromachining and Microfabrication Process Technology XI*, 61090A, 2006, vol. 6109, p. 61090A–61090A–8.

- [46] a. Sharon, a. Bilsing, G. Lewis, and X. Z. X. Zhang, "Manufacturing of 3D microstructures using novel UPSAMS process (ultra precision manufacturing of self-assembled micro systems)," *Sixt. Annu. Int. Conf. Micro Electro Mech. Syst. 2003. MEMS-03 Kyoto. IEEE*, pp. 542–545, 2003.
- [47] W. Pfleging, W. Bernauer, T. Hanemann, and M. Torge, "Rapid fabrication of microcomponents - UV-laser assisted prototyping, laser micro-machining of mold inserts and replication via photomolding," *Microsyst. Technol.*, vol. 9, no. 1–2, pp. 67–74, 2003.
- [48] C. L. Kuo, J. D. Huang, and H. Y. Liang, "Fabrication of 3D Metal Microstructures Using a Hybrid Process of Micro-EDM and Laser Assembly," *Int. J. Adv. Manuf. Technol.*, vol. 21, no. 10–11, pp. 796–800, 2003.
- [49] J.-W. Chen and J. M. Zybko, "Diode laser bonding of planar microfluidic devices, MOEMS, bioMEMS, diagnostic chips, and microarrays," in *Proceedings SPIE 5718, Microfluidics, BioMEMS, and Medical Microsystems III*, 2005, vol. 5718, pp. 92–98.
- [50] A. J. Krejcie, S. G. Kapoor, and R. E. DeVor, "A hybrid process for manufacturing surgical-grade knife blade cutting edges from bulk metallic glass," *J. Manuf. Process.*, vol. 14, no. 1, pp. 26–34, Jan. 2012.
- [51] S. H. Ahn, D. M. Chun, and C. S. Kim, "Nanoscale hybrid manufacturing process by nano particle deposition system (NPDS) and focused ion beam (FIB)," *CIRP Ann. - Manuf. Technol.*, vol. 60, no. 1, pp. 583–586, 2011.
- [52] B. X. Jia, Z. L. Wang, F. Q. Hu, X. H. Li, and W. S. Zhao, "Research on Multifunctional Micro Machining Equipment," *Mater. Sci. Forum*, vol. 471–472, pp. 37–42, 2004.
- [53] L. B. Zhou, Y. Yaguchi, T. Fujii, J. Shimizu, and H. Eda, "Development of a Multifunctional Micro-Machining System and its Applications," *Key Eng. Mater.*, vol. 238–239, pp. 3–8, 2003.
- [54] Y.-S. Liao, S.-T. Chen, and C.-S. Lin, "Development of A Multi-function High Precision Tabletop CNC Machine for Making Micro Parts (Advanced machine tool)," *Proc. Int. Conf. Lead. Edge Manuf. 21st century LEM21*, vol. 2005, no. 2, pp. 485–490, Oct. 2005.
- [55] M. Rahman, H. S. Lim, K. S. Neo, A. Senthil Kumar, Y. S. Wong, and X. P. Li, "Tool-

- based nanofinishing and micromachining," *J. Mater. Process. Technol.*, vol. 185, no. 1–3, pp. 2–16, Apr. 2007.
- [56] Y. B. Bang, K. M. Lee, and S. Oh, "5-Axis Micro Milling Machine for Machining Micro Parts," *Int. J. Adv. Manuf. Technol.*, vol. 25, no. 9–10, pp. 888–894, 2005.
- [57] M. Arentoft and N. A. Paldan, "Production equipment for manufacturing of micro metal components," in *Proceedings of the 9th International ESAFORM Conference on Material Forming*, 2006, pp. 579–582.
- [58] Y. Qin, Y. Ma, C. Harrison, A. Brockett, M. Zhou, J. Zhao, F. Law, A. Razali, R. Smith, and J. Eguia, "Development of a new machine system for the forming of micro-sheet-products," *Int. J. Mater. Form.*, vol. 1, no. 1, pp. 475–478, 2008.
- [59] C. Hartl, J. Lungershausen, J. Equia, L. Uriate, and P. Lopes Garcia, "Micro hydroforming process and machine system for miniature/micro products," in *Proceedings of International Conference 7th Euspen, Bremen 2*, 2007, pp. 69–72.
- [60] G.-Y. Kim, J. Ni, and M. Koç, "Modeling of the Size Effects on the Behavior of Metals in Microscale Deformation Processes," *J. Manuf. Sci. Eng.*, vol. 129, no. 3, p. 470, 2007.
- [61] W. Sylwestrowicz and E. O. Hall, "The Deformation and Ageing of Mild Steel," *Proc. Phys. Soc. Sect. B*, vol. 64, no. 9, pp. 747–753, 2002.
- [62] N. J. Petch, "The Cleavage Strength of Polycrystals," *J. Iron Steel Inst.*, vol. 174, pp. 25–28, 1953.
- [63] M. Koç and S. Mahabunphachai, "Feasibility investigations on a novel micro-manufacturing process for fabrication of fuel cell bipolar plates: Internal pressure-assisted embossing of micro-channels with in-die mechanical bonding," *J. Power Sources*, vol. 172, no. 2, pp. 725–733, Oct. 2007.
- [64] N. Hansen, "The effect of grain size and strain on the tensile flow stress of aluminium at room temperature," *Acta Metall.*, vol. 25, no. 8, pp. 863–869, Aug. 1977.
- [65] L. V. Raulea, A. M. Goijaerts, L. E. Govaert, and F. P. T. Baaijens, "Size effects in the processing of thin metal sheets," *J. Mater. Process. Technol.*, vol. 115, no. 1, pp. 44–48, Aug. 2001.
- [66] J. . Michel and P. Picart, "Size effects on the constitutive behaviour for brass in sheet

- metal forming,” *J. Mater. Process. Technol.*, vol. 141, no. 3, pp. 439–446, Nov. 2003.
- [67] T. A. Kals and R. Eckstein, “Miniaturization in sheet metal working,” *J. Mater. Process. Technol.*, vol. 103, no. 1, pp. 95–101, Jun. 2000.
- [68] S. Miyazaki, H. Fujita, and H. Hiraoka, “Effect of specimen size on the flow stress of rod specimens of polycrystalline Cu-Al alloy,” *Scr. Metall.*, vol. 13, no. 6, pp. 447–449, Jun. 1979.
- [69] R. M. Onyancha and B. L. Kinsey, “Investigation of Size Effects on Process Models for Plane Strain Microbending,” in *ASME 2006 International Manufacturing Science and Engineering Conference, Manufacturing Science and Engineering, Parts A and B, Ypsilanti, Michigan, USA*, 2006, pp. 257–266.
- [70] J. T. Black and R. A. Kohser, “Powder Metallurgy,” in *DeGarmo’s Materials and Processes in Manufacturing*, Wiley, 2011, p. 461.
- [71] R. K. Raiput, “Powder Metallurgy,” in *A Textbook of Manufacturing Technology: Manufacturing Process*, Laxmi Publications (P) LTD, 2007, pp. 219–220.
- [72] J. W. Newkirk and S. N. Thakur, “Heat Treatment of Powder Metallurgy Steel Components,” in *Steel Heat Treatment*, CRC Press, 2006, pp. 741–786.
- [73] “Powder Metallurgy - Conventional Processing,” *European Powder Metallurgy Association (EPMA)*. [Online]. Available: <http://www.epma.com/powder-metallurgy-conventional-processing>. [Accessed: 04-Oct-2016].
- [74] E. P De Garmo, J. T Black, and R. A Kohser, “Powder Metallurgy,” in *DeGarmo’s Materials and Processing in Manufacturing*, 11th ed., Hoboken, NJ : John Wiley & Sons, 2013, pp. 481–506.
- [75] The European Powder Metallurgy Association, “Powder Metallurgy - Sintering Temperatures for Some Common Metals,” *The European Powder Metallurgy Association*, 2013. [Online]. Available: <http://www.azom.com/article.aspx?ArticleID=1727>. [Accessed: 18-Sep-2015].
- [76] O. A. Troitskii, “Electromechanical Effect in Metals,” *ZhETF Pis ma Redaktsiiu*, vol. 10, pp. 18–22, 1969.
- [77] G. Tang, J. Zhang, M. Zheng, J. Zhang, W. Fang, and Q. Li, “Experimental study of

- electroplastic effect on stainless steel wire 304L," *Mater. Sci. Eng. A*, vol. 281, no. 1–2, pp. 263–267, Apr. 2000.
- [78] G. Tang, M. Zheng, Y. Zhu, J. Zhang, W. Fang, and Q. Li, "The application of the electro-plastic technique in the cold-drawing of steel wires," *J. Mater. Process. Technol.*, vol. 84, no. 1–3, pp. 268–270, Dec. 1998.
- [79] M. S. Siopis, "Investigation of electrical-assisted forming at the microscale," University of New Hampshire, Ann Arbor, 2009.
- [80] J. Mai, L. Peng, X. Lai, and Z. Lin, "Electrical-assisted embossing process for fabrication of micro-channels on 316L stainless steel plate," *J. Mater. Process. Technol.*, vol. 213, no. 2, pp. 314–321, Feb. 2013.
- [81] C. Ross and J. T. Roth, "The Effects of DC Current on the Tensile Properties of Metals," in *ASME 2005 International Mechanical Engineering Congress and Exposition Materials*, 2005, pp. 363–372.
- [82] R. E. Reed-Hill, *Physical Metallurgy Principles*, 2nd Ed. PWS Kent, 1973.
- [83] T. a. Perkins, T. J. Kronenberger, and J. T. Roth, "Metallic Forging Using Electrical Flow as an Alternative to Warm/Hot Working," *J. Manuf. Sci. Eng.*, vol. 129, no. 1, p. 84, 2007.
- [84] L. Yao, C. Hong, G. Yunqou, and H. Xinbin, "Effect of Electric Current Pulse On Superplastic of Aluminum Alloy 7475," *Trans. Nonferrous Met. Soc. China*, vol. 6, no. 1, pp. 77–80, 1996.
- [85] O. A. Troitskii, "Anisotropy of the Effect of Electron-Beam and α -Irradiation on the Deformation Process of Zinc Single Crystals in the Brittle State," *Akad. Nauk SSSR* 148, pp. 332–334, 1963.
- [86] H. Conrad, "Electroplasticity in metals and ceramics," *Mater. Sci. Eng. A*, vol. 287, no. 2, pp. 276–287, Aug. 2000.
- [87] G. Tang, J. Zhang, Y. Yan, H. Zhou, and W. Fang, "The engineering application of the electroplastic effect in the cold-drawing of stainless steel wire," *J. Mater. Process. Technol.*, vol. 137, no. 1–3, pp. 96–99, Jun. 2003.
- [88] C. D. Ross, D. B. Irvin, and J. T. Roth, "Manufacturing Aspects Relating to the Effects

- of Direct Current on the Tensile Properties of Metals,” *J. Eng. Mater. Technol.*, vol. 129, no. 2, p. 342, 2007.
- [89] J. S. Andrawes, T. J. Kronenberger, T. a. Perkins, J. T. Roth, and R. L. Warley, “Effects of DC Current on the Mechanical Behavior of AlMg1SiCu,” *Mater. Manuf. Process.*, vol. 22, no. 1, pp. 91–101, 2007.
- [90] J. T. Roth, I. Loker, D. Mauck, M. Warner, S. F. Golovashchenko, and A. Krause, “Enhanced Formability of 5754 Aluminum Sheet Metal Using Electric Pulsing,” in *Society of Manufacturing Engineers (Technical Papers)*, 2008, pp. 405–412.
- [91] V. V. Stolyarov, “Deformability and nanostructuring of TiNi shape-memory alloys during electroplastic rolling,” *Mater. Sci. Eng. A*, vol. 503, no. 1–2, pp. 18–20, Mar. 2009.
- [92] M. S. Siopis and B. L. Kinsey, “Experimental Investigation of Grain and Specimen Size Effects During Electrical-Assisted Forming,” *J. Manuf. Sci. Eng.*, vol. 132, no. 2, p. 21004, 2010.
- [93] Y. Lu, T. Qu, P. Zeng, L. Lei, G. Fang, and J. Sun, “The influence of electroplastic rolling on the mechanical deformation and phase evolution of Bi-2223/Ag tapes,” *J. Mater. Sci.*, vol. 45, no. 13, pp. 3514–3519, 2010.
- [94] S. Maki, Y. Harada, K. Mori, and H. Makino, “Application of resistance heating technique to mushy state forming of aluminium alloy,” *J. Mater. Process. Technol.*, vol. 125–126, pp. 477–482, Sep. 2002.
- [95] K. Mori, S. Maki, and Y. Tanaka, “Warm and Hot Stamping of Ultra High Tensile Strength Steel Sheets Using Resistance Heating,” *CIRP Ann. - Manuf. Technol.*, vol. 54, no. 1, pp. 209–212, Jan. 2005.
- [96] K. Mori, S. Saito, and S. Maki, “Warm and hot punching of ultra high strength steel sheet,” *CIRP Ann. - Manuf. Technol.*, vol. 57, no. 1, pp. 321–324, 2008.
- [97] J. Yanagimoto and R. Izumi, “Continuous electric resistance heating—Hot forming system for high-alloy metals with poor workability,” *J. Mater. Process. Technol.*, vol. 209, no. 6, pp. 3060–3068, Mar. 2009.
- [98] C. M. Dzialo, M. S. Siopis, B. L. Kinsey, and K. J. Weinmann, “Effect of current density and zinc content during electrical-assisted forming of copper alloys,” *CIRP Ann. -*

Manuf. Technol., vol. 59, no. 1, pp. 299–302, 2010.

- [99] K. Mori and Y. Okuda, "Tailor die quenching in hot stamping for producing ultra-high strength steel formed parts having strength distribution," *CIRP Ann. - Manuf. Technol.*, vol. 59, no. 1, pp. 291–294, 2010.
- [100] K. Mori, T. Maeno, and Y. Fukui, "Spline forming of ultra-high strength gear drum using resistance heating of side wall of cup," *CIRP Ann. - Manuf. Technol.*, vol. 60, no. 1, pp. 299–302, 2011.
- [101] M. Eriksson, Z. Shen, and M. Nygren, "Fast densification and deformation of titanium powder," *Powder Metall.*, vol. 48, no. 3, pp. 231–236, 2005.
- [102] R. Chaudhari and R. Bauri, "Microstructure and Mechanical Properties of Titanium Processed by Spark Plasma Sintering (SPS)," *Metallogr. Microstruct. Anal.*, vol. 3, no. 1, pp. 30–35, 2014.
- [103] K. Asaoka and M. Kon, "Sintered porous titanium and titanium alloys as advanced biomaterials," *Thermec 2003 Processing and Manufacturing of Advanced Materials*, vol. 426–432, no. 4. Department of Dental Engineering, School of Dentistry, University of Tokushima, 3 Kuramoto-cho, Tokushima 770-8504, Japan, pp. 3079–3084, 2003.
- [104] J. Onagawa, T. Goto, O. Ise, N. Ishii, T. Horikawa, and K. Sawada, "Corrosion Resistance of Titanium-Platinum Alloy Prepared by Spark Plasma Sintering," *Mater. Trans. JIM*, vol. 37, no. 11, pp. 1699–1703, 1996.
- [105] Y. F. Yang, H. Imai, K. Kondoh, and M. Qian, "Comparison of spark plasma sintering of elemental and master alloy powder mixes and prealloyed Ti-6Al-4V powder," *Int. J. Powder Metall.*, vol. 50, no. 1, pp. 41–47, 2014.
- [106] S. . Wang, L. . Chen, Y. . Kang, M. Niino, and T. Hirai, "Effect of plasma activated sintering (PAS) parameters on densification of copper powder," *Mater. Res. Bull.*, vol. 35, no. 4, pp. 619–628, 2000.
- [107] T. S. Srivatsan, B. G. Ravi, A. S. Naruka, L. Riester, S. Yoo, and T. S. Sudarshan, "Microstructure and hardness of copper powder consolidated by plasma pressure compaction," *J. Mater. Eng. Perform.*, vol. 10, no. 4, pp. 449–455, 2001.
- [108] T. S. Srivatsan, B. G. Ravi, A. S. Naruka, L. Riester, S. Yoo, and T. S. Sudarshan, "A

- study of microstructure and hardness of bulk copper sample obtained by consolidating nanocrystalline powders using plasma pressure compaction," *Mater. Sci. Eng. A*, vol. 311, no. 1, pp. 22–27, 2001.
- [109] Z. J. Zhou and Y. S. Kwon, "Fabrication of W–Cu composite by resistance sintering under ultra-high pressure," *J. Mater. Process. Technol.*, vol. 168, no. 1, pp. 107–111, 2005.
- [110] C. H. Liu and P. W. Kao, "Microstructure and mechanical properties of resistance sintered titanium," *Scr. Metall. Mater.*, vol. 24, no. 12, pp. 2279–2284, Dec. 1990.
- [111] M. Zadra, F. Casari, L. Girardini, and A. Molinari, "Microstructure and mechanical properties of cp-titanium produced by spark plasma sintering," *Powder Metall.*, vol. 51, no. 1, pp. 59–65, Mar. 2008.
- [112] L. Liu, G. Liu, Y. Xiong, J. Chen, C. Kang, X. Huang, and Y. Tian, "Fabrication of Fresnel zone plates with high aspect ratio by soft X-ray lithography," *Microsyst. Technol.*, vol. 14, no. 9–11, pp. 1251–1255, 2008.
- [113] J. R. Serrano and L. M. Phinney, "Displacement and thermal performance of laser-heated asymmetric MEMS actuators," *J. Microelectromechanical Syst.*, vol. 17, no. 1, pp. 166–174, 2008.
- [114] K. Feng, M. Hong, Y. Yang, and W. Wang, "Combustion synthesis of VC/Fe composites under the action of an electric field," *Int. J. Refract. Met. Hard Mater.*, vol. 27, no. 5, pp. 852–857, Sep. 2009.
- [115] M. Demuyne, J.-P. Erauw, O. Van der Biest, F. Delannay, and F. Cambier, "Densification of alumina by SPS and HP: A comparative study," *J. Eur. Ceram. Soc.*, vol. 32, no. 9, pp. 1957–1964, Jul. 2012.
- [116] Z. Shen, M. Johnsson, Z. Zhao, and M. Nygren, "Spark Plasma Sintering of Alumina," *J. Am. Ceram. Soc.*, vol. 85, no. 8, pp. 1921–1927, 2002.
- [117] M. P. Harmer and R. J. Brook, "Fast firing-microstructure benefits," *J. Br. Ceram. Soc.*, vol. 80, no. 5, pp. 147–148, 1981.
- [118] M. Zulkipli, Y. Qin, K. Huang, H. Hijji, Y. Zhao, and J. Zhao, "Forming of titanium and titanium alloy miniature-cylinders by electrical-field activated powder sintering and

forming,” in *4th International Conference on New Forming Technology (ICNFT 2015): MATEC Web of Conferences*, 2015, vol. 21.

- [119] M. Karttunen, P. Ruuskanen, and J. Enqvist, “Electrically Conductive Composite Powders and Compounds Produced with Solid State Synthesis,” *Mater. Manuf. Process.*, vol. 20, no. 5, pp. 887–892, 2005.
- [120] D. V. Quach, H. Avila-Paredes, S. Kim, M. Martin, and Z. A. Munir, “Pressure effects and grain growth kinetics in the consolidation of nanostructured fully stabilized zirconia by pulsed electric current sintering,” *Acta Mater.*, vol. 58, no. 15, pp. 5022–5030, Sep. 2010.
- [121] Z. A. Munir, “The effect of external electric fields on the nature and properties of materials synthesized by self-propagating combustion,” *Mater. Sci. Eng. A*, vol. 287, no. 2, pp. 125–137, Aug. 2000.
- [122] F. Wakai and Y. Shinoda, “Anisotropic sintering stress for sintering of particles arranged in orthotropic symmetry,” *Acta Mater.*, vol. 57, no. 13, pp. 3955–3964, Aug. 2009.
- [123] W. Chen, *Gleeble System and Application*, 1st ed. New York, NY, USA: Gleeble System School, 1998.
- [124] F. Li, J. Pan, O. Guillon, and A. Cocks, “Predicting sintering deformation of ceramic film constrained by rigid substrate using anisotropic constitutive law,” *Acta Mater.*, vol. 58, no. 18, pp. 5980–5988, Oct. 2010.
- [125] N. Beri, S. Maheshwari, C. Sharma, and A. Kumar, “Technological Advancement in Electrical Discharge Machining with Powder Metallurgy Processed Electrodes: A Review,” *Mater. Manuf. Process.*, vol. 25, no. 10, pp. 1186–1197, 2010.
- [126] D. Lu, Y. Yang, Y. Qin, and G. Yang, “Effect of particle size and sintering temperature on densification during coupled multifield-activated microforming,” *J. Mater. Res.*, vol. 27, no. 20, pp. 2579–2586, 2012.
- [127] P. Murray, E. P. Rodgers, and A. E. Williams, “Practical and theoretical aspects of the hot pressing of refractory oxides,” *Trans. J. Br. Ceram. Soc.*, vol. 53, pp. 474–509, 1954.

- [128] K. Huang, Y. Yang, Y. Qin, and G. Yang, "316 L Stainless Steel Powder Densification during the Coupled Multi-Fields Activated Micro-Forming," *Mater. Manuf. Process.*, vol. 28, no. 2, pp. 183–188, Feb. 2013.
- [129] K. Huang, Y. Yang, Y. Qin, and G. Yang, "Densification behavior of copper powder during the coupled multi-physics fields-activated microforming," *Int. J. Adv. Manuf. Technol.*, vol. 69, no. 9–12, pp. 2651–2657, Aug. 2013.
- [130] J. Liu, Y. Yang, K. Feng, and D. Lu, "Study on the effect of current on reactive sintering of the W–C–Co mixture under an electric field," *J. Alloys Compd.*, vol. 476, no. 1–2, pp. 207–212, May 2009.
- [131] A. K. Du, G. Yang, D. Lu, Y. Qin, and Y. Yang, "Densification of MnZn Ferrite Sintered under Multi-Physical Field Coupling," *Appl. Mech. Mater.*, vol. 271–272, pp. 212–217, 2012.
- [132] A. Du, Y. Yang, Y. Qin, and G. Yang, "Effects of Heating Rate and Sintering Temperature on 316 L Stainless Steel Powders Sintered Under Multiphysical Field Coupling," *Mater. Manuf. Process.*, vol. 28, no. 1, pp. 66–71, 2012.
- [133] D. Lu, Y. Yang, Y. Qin, and G. Yang, "Forming Microgears by Micro-FAST Technology," *J. Microelectromechanical Syst.*, vol. 22, no. 3, pp. 708–715, Jun. 2013.
- [134] "CES EduPack 2013 Version 12.2.13," *Granta Design Limited*. 2013.
- [135] H. W. Zhang, R. Gopalan, T. Mukai, and K. Hono, "Fabrication of bulk nanocrystalline Fe–C alloy by spark plasma sintering of mechanically milled powder," *Scr. Mater.*, vol. 53, no. 7, pp. 863–868, Oct. 2005.
- [136] R. Nicula, F. Turquier, M. Stir, V. Y. Kodash, J. R. Groza, and E. Burkel, "Quasicrystal phase formation in Al–Cu–Fe nanopowders during field-activated sintering (FAST)," *J. Alloys Compd.*, vol. 434–435, pp. 319–323, May 2007.
- [137] "Abaqus Theory Manual: 6.7.2 Coupled Thermal-Electrical Analysis," *Dassault System Simulia*. 2010.
- [138] T. Ogasawara, Y. Hirano, and A. Yoshimura, "Coupled thermal-electrical analysis for carbon fiber/epoxy composites exposed to simulated lightning current," *Compos. Part A Appl. Sci. Manuf.*, vol. 41, no. 8, pp. 973–981, 2010.

- [139] H. R. Ogden and R. I. Jaffee, "The effects of carbon, oxygen and nitrogen on the mechanical properties of titanium and titanium alloys," Titanium Metallurgical Laboratory, Battelle Memorial Institute, Columbus, Ohio, United States, TML-20, 1955.
- [140] M. Chu, I. P. Jones, and X. Wu, "Effect of Carbon on Microstructure and Mechanical Properties of a Eutectoid β Titanium Alloy," *J. Mater. Eng. Perform.*, vol. 14, no. 6, pp. 735–740, 2005.

